

## **APPENDIX G**

### **Analytical Data QA/QC Report and USACE-prepared Chemical Data Quality Assurance Report**

**DATA VALIDATION SUMMARY REPORT**

**for samples collected from**

**BROWN FIELD BOMBING RANGE**

**San Diego County, California**

Data Validation by: Katherine LaPierre

Parsons - Austin

**INTRODUCTION**

The following data validation summary report covers soil samples collected from the Brown Field Bombing Range in San Diego County, California on June 25 and 26, 2007. Samples were logged in under the following Sample Delivery Group (SDG):

D7F270299

The samples in this SDG were analyzed for explosives and metals. The table below details the requested parameters for each sample. The field quality control (QC) samples associated with this SDG included one matrix spike/matrix spike duplicate (MS/MSD) pair and one field duplicate. The field QC samples were analyzed for the same parameters as the associated parent sample.

All samples were collected by Parsons. All analyses were performed by TestAmerica (formerly STL) in Denver, Colorado following the procedures outlined in the Standard Subcontract and the Sampling and Analysis Plan (SAP) Addendum for the Southwest Region. The samples in this SDG were shipped to the laboratory in one cooler. The cooler was received by laboratory at a temperature of 0.6°C, which was below the 2-6° C range recommended by the Work Plan. However, all samples were received in tact (not frozen) so data quality was not affected and no corrective action was necessary.

**SAMPLE IDS AND REQUESTED PARAMETERS**

Sample ID	Matrix	Explosives	Metals	Comments
BFB-C-SS-03-01	S	X	X	Ambient
BFB-B-SS-03-02	S	X	X	
BFB-C-SS-03-03	S	X	X	Ambient, MS/MSD
BFB-A-SS-03-04	S	X	X	
BFB-A-SS-03-05	S	X	X	
BFB-A-SS-03-06	S	X	X	
BFB-A-SS-03-07	S	X	X	
BFB-B-SS-03-08	S	X	X	
BFB-C-SS-03-09	S	X	X	
BFB-C-SS-03-10	S	X	X	
BFB-C-SS-03-12	S	X	X	FD of BFB-C-SS-03-03

S = Soil

**EXTRACTION, ANALYTICAL, AND REPORTING DETAILS**

Parameter	Matrix	Extraction Method	Analytical Method	Dry Weight / Wet Weight	Units
Explosives	Soil	SW8330A	SW8321A	Dry	µg/kg
ICP-AES Metals	Soil	SW3050B	SW6010B	Dry	mg/kg
ICP/MS Metals	Soil	SW3050B	SW6020	Dry	mg/kg
Mercury	Soil	SW7471A	SW7471A	Dry	mg/kg

ICP-AES = Inductively Coupled Plasma Atomic Emission Spectroscopy

ICP/MS = Inductively Coupled Plasma Mass Spectroscopy

**EVALUATION CRITERIA**

The data submitted by the laboratory has been reviewed and verified following the guidelines outlined in the PPSAP (consisting of the Field Sampling Plan [FSP] and the Quality Assurance Project Plan [QAPP]) for the MMRP SI Program, prepared by the USACE Military Munitions Center of Expertise (MM CX). Information reviewed in the data packages included sample results; field and laboratory quality control results; calibrations; case narratives; raw data; cooler receipt forms, and chain-of-custody (COC) forms. The analyses and findings presented in this report are based on the reviewed information, and whether guidelines in the Work Plan were met.

Due to the flagging requirements of the electronic data deliverable (EDD) software, Automatic Data Review (ADR), the following rules were applied for flagging the data:

If an analyte was detected in the method blank, the associated sample concentrations were examined. If the analyte was detected in a sample at a concentration similar to that found in the blank (five times the blank concentration for most analytes, or ten times the blank concentration for common laboratory contaminants), the reporting limit for that analyte was raised to the detected level and the result was flagged “U” for that particular sample.

Approval was received from a United States Army Corps of Engineers (USACE) chemist for the laboratory to use the historically developed control limits for explosives, as listed in the table below.

Analyte	Accuracy Control Limits for Soil	Maximum RPD
HMX	53-115	30
RDX	70-121	30
1,3,5-Trinitrobenzene	47-131	30
1,3-dinitrobenzene	69-128	30
Nitrobenzene	59-150	30
Tetryl	10-160	30
Nitroglycerin	32-135	30
2,4,6-Trinitrotoluene	58-130	30
4-Amino-2,6-dinitrotoluene	60-133	30
2-Amino-4,6-dinitrotoluene	53-141	30
2,4-Dinitrotoluene	61-128	30
2,6-Dinitrotoluene	59-134	30
3-Nitrotoluene	51-153	30
PETN	28-178	30
2-Nitrotoluene	55-147	30
4-Nitrotoluene	65-146	30

For metals, the accuracy control limits are 80-120% for the LCS and MS/MSD, and the maximum allowable RPD for the precision of the MS/MSD is 20%.

The field duplicate tolerances ( $RPD \leq 70$  for soil) were approved by Deborah Walker and Brian Jordan for this site.

## EXPLOSIVES

### General

The explosives portion of this SDG consisted of eleven (11) soil samples. The samples were collected on June 25 and 26, 2007 and were analyzed for the full list of explosives as specified in the Work Plan.

The explosives analyses were performed according to USEPA SW846 Method 8321A. All samples in this SDG were analyzed following the procedures outlined in the laboratory Standard Operating Procedure (SOP). All samples were prepared and analyzed within the holding time required by the method.

The explosives samples were extracted and analyzed in one batch (#7183436) under a single initial calibration (ICAL).

## Accuracy

Accuracy was evaluated using the percent recovery (%R) obtained from the laboratory control spike (LCS) sample, MS/MSD samples, and the surrogate spikes. Sample BFB-C-SS-03-03 was designated for MS/MSD analyses for this SDG.

All LCS and surrogate spike recoveries were within acceptance criteria.

All MS/MSD recoveries were within acceptance criteria, except for the following:

Analyte	MS %R	MSD %R	Criteria
1,3,5-Trinitrobenzene	(118)	132	47-131%
2,4,6-Trinitrotoluene	(115)	132	58-130%

( ) indicates the recovery met criteria.

Both non-compliant MSD recoveries were considered minor exceedances. No corrective action was deemed necessary because the MSD recoveries were only one or two percent high and all sample results were non-detect. The “J” flags applied by the laboratory to the parent sample results for these compounds were removed.

## Precision

Precision was evaluated using the relative percent difference (RPD) obtained from the MS/MSD concentrations. Precision was further assessed by comparing the field duplicate analyte results. Sample BFB-C-SS-03-12 was collected as a field duplicate of sample BFB-C-SS-03-03.

All MS/MSD RPDs were within acceptance criteria.

All analytes were non-detect in both the parent and field duplicate samples.

## Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing the COC procedures to those described in the Work Plan;
- Comparing actual analytical procedures to those described in the Work Plan;
- Evaluating holding times; and
- Examining laboratory blanks for cross contamination of samples during analysis.

The samples in this SDG were analyzed following the COC and the analytical procedures described in the Work Plan. All samples were prepared and analyzed within the holding time required by the method and the Work Plan.

- All initial calibration criteria were met.
- All secondary source verification criteria were met.
- All initial and continuing calibration verification criteria were met.
- The MDL studies for soil were conducted on April 4, 2006 and these samples were analyzed on July 9 and 10, 2007. The laboratory performed a new MDL study on January 26, 2007. However, several analytes did not meet the criteria

that the MDL be one-third the reporting limit (RL), so the laboratory is currently in the process of repeating the MDL study using a lower spike amount.

One method blank was associated with this SDG. The method blank was non-detect for all target explosives.

### **Completeness**

Completeness has been evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All explosives results for the samples in this SDG were considered usable. Thus, the completeness for the explosives portion of this SDG is 100%, which meets the minimum acceptance criteria of 95%.

### **ICP-AES Metals**

#### **General**

The ICP-AES portion of this SDG consisted of eleven (11) soil samples collected on June 25 and 26, 2007. The samples were analyzed for aluminum, calcium, iron, magnesium, potassium, sodium, strontium, and titanium.

The ICP-AES metals analyses were performed using USEPA SW846 Method 6010B. The samples in this SDG were analyzed following the procedures outlined in the Work Plan. All samples were prepared and analyzed within the holding time required by the method and the Work Plan.

The ICP-AES metals samples were digested and analyzed in one batch (#7181055) under a single ICAL.

#### **Accuracy**

Accuracy was evaluated using the percent recovery obtained from the LCS samples and the MS/MSD samples. Sample BFB-C-SS-03-03 was designated for MS/MSD analyses for this SDG.

All LCS recoveries were within acceptance criteria.

All MS/MSD recoveries were within acceptance criteria, except for the following:

#### **BFB-C-SS-03-03**

<b>Analyte</b>	<b>MS %R</b>	<b>MSD %R</b>	<b>Criteria</b>
Aluminum	2350	1830	80 – 120%
Iron	1620	361	
Titanium	181	150	

It should be noted that the parent sample concentrations for aluminum and iron were significantly greater than (more than thirty times) the amount spiked, resulting in the anomalous recoveries. The aluminum, iron, and titanium results were flagged “J” in the parent sample, in accordance with the PSAP.

## Precision

Precision was evaluated using the RPD obtained from the MS/MSD concentrations. Precision was further assessed by comparing the field duplicate analyte results. Sample BFB-C-SS-03-12 was collected as a field duplicate of sample BFB-C-SS-03-03.

All MS/MSD RPDs were within acceptance criteria.

All field duplicate RPDs met criteria for those metals detected above the RL in both the parent and field duplicate samples, as follows:

BFB-C-SS-03-03				
Analyte	Parent (mg/kg)	FD (mg/kg)	RPD	Criteria
Aluminum	6900	6800	1.5	RPD $\leq$ 70
Calcium	1100	1200	8.7	
Iron	9600	8900	7.6	
Magnesium	1400	1400	0	
Potassium	1500	1400	6.9	
Strontium	11	11	0	
Titanium	200	200	0	

## Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing the COC procedures to those described in the Work Plan;
- Comparing actual analytical procedures to those described in the Work Plan;
- Evaluating preservation and holding times; and
- Examining laboratory blank for cross contamination of samples during analysis.

The samples in this SDG were analyzed following the COC and the analytical procedures described in the Work Plan. All samples were prepared and analyzed within the holding time required by the method and the Work Plan.

- All initial calibration criteria were met.
- All second source verification criteria were met. The initial calibration verification samples were prepared using a secondary source.
- All continuing calibration verification criteria were met.
- All interference check criteria were met.
- All RL check standard criteria were met.
- A dilution test (DT) was performed on sample BFB-C-SS-03-03 for all metals detected in the parent sample at a concentration of 50 times the MDL or greater. All metals met criteria with the exception of those in bold below:

Metal	%D	Criteria
-------	----	----------

<b>Aluminum</b>	<b>13</b>	%D ≤10
<b>Calcium</b>	<b>13</b>	
Iron	9.3	
<b>Magnesium</b>	<b>13</b>	
<b>Strontium</b>	<b>12</b>	
Titanium	10	

- A post digestion spike (PDS) was performed on the same sample as the DT. The PDS was only applicable for the metals in bold above and those metals not listed above. The PDS met criteria for all applicable metals, with the exception of aluminum, as follows:

<b>Metal</b>	<b>%R</b>	<b>Criteria</b>
<b>Aluminum</b>	<b>0</b>	75 – 125%
Calcium	88	
Potassium	87	
Magnesium	87	
Sodium	91	
Strontium	89	

Aluminum was flagged “J” as estimated in all samples in this SDG due to the non-compliant DT and PDS results.

One method blank and several calibration blanks were analyzed in association with the ICP-AES analyses in this SDG. All blanks were compliant.

### **Completeness**

Completeness has been evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All ICP-AES metals results for the samples in this SDG were considered usable. The completeness for the ICP metals portion of this SDG is 100%, which meets the minimum acceptance criteria of 95%.

## **TOTAL ICP/MS METALS**

### **General**

The total ICP/MS portion of this SDG consisted of eleven (11) soil samples collected on June 25 and 26, 2007. The samples were analyzed for antimony, arsenic, barium, beryllium, cadmium, chromium, cobalt, copper, lead, manganese, molybdenum, nickel, selenium, silver, thallium, vanadium, and zinc by USEPA SW846 Method 6020.

The samples were analyzed following the procedures outlined in the Work Plan. All samples were prepared and analyzed within the holding time required by the method and the Work Plan.

The total ICP/MS metals samples were digested and analyzed in one batch (#7181046) under a single ICAL.



### Accuracy

Accuracy was evaluated using the percent recovery obtained from the LCS samples and the MS/MSD samples. Sample BFB-C-SS-03-03 was designated for MS/MSD analyses for this SDG.

All LCS recoveries were within acceptance criteria.

All analyte recoveries were within acceptance criteria for the MS/MSD, with the following exceptions:

Metal	MS %R	MSD %R	Criteria
Antimony	5.0	4.5	80-120%
Arsenic	71	(86)	
Barium	0	233	
Cobalt	75	(86)	
Copper	76	(93)	
Lead	125	155	
Manganese	0	147	
Molybdenum	59	59	
Nickel	77	(88)	
Selenium	71	72	
Vanadium	71	197	
Zinc	57	157	

( ) indicates the recovery met criteria.

It should be noted that the parent sample concentrations for barium and manganese were significantly greater than (more than fifteen times) the amount spiked, resulting in the anomalous recoveries. No corrective action was deemed necessary for cobalt, copper, or nickel because these metals met criteria in the MSD and were only slightly (5% or less) below criteria in the MS. The “J” flags applied to the parent sample results for these metals were removed. All other non-compliant metals were flagged “J” if detected or “UJ” if non-detect in the parent sample in accordance with the PSAP.

### Precision

Precision was evaluated using the RPD obtained from the MS/MSD concentrations. Precision was further assessed by comparing the field duplicate analyte results. Sample BFB-C-SS-03-12 was collected as a field duplicate of sample BFB-C-SS-03-03.

All MS/MSD RPDs were within acceptance criteria with the exception of vanadium. The RPD for vanadium exceeded criteria ( $RPD \leq 20$ ) at 21. This metal was previously flagged “J” in the parent sample due to the non-compliant MS/MSD results, so no additional corrective action was necessary.

All metals detected above the RL in both the parent and field duplicate samples met criteria, as follows:

BFB-C-SS-03-03				
Metal	Parent Conc.	FD Conc.	RPD	Criteria

	(mg/kg)	(mg/kg)		
Arsenic	2.3	2.4	4.3	RPD $\leq$ 70
Barium	64	63	1.6	
Beryllium	0.26	0.26	0	
Chromium	5.6	5.8	3.5	
Cobalt	3.1	3.2	3.2	
Copper	4.8	4.6	4.3	
Lead	9.8	9.3	5.2	
Manganese	240	240	0	
Nickel	2.7	2.6	3.8	
Selenium	0.57	0.56	1.8	
Thallium	0.12	0.13	8.0	
Vanadium	19	20	5.1	
Zinc	20	19	5.1	

### Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing the COC procedures to those described in the Work Plan;
- Comparing actual analytical procedures to those described in the Work Plan;
- Evaluating preservation and holding times; and
- Examining laboratory blanks for cross contamination of samples during analysis.

The samples in this SDG were analyzed following the COC and the analytical procedures described in the Work Plan. The samples were prepared and analyzed within the holding times required by the method.

- All instrument tune criteria were met.
- All initial calibration criteria were met.
- All metals met criteria in the RL check standards.
- All second source criteria were met. The ICV sample was prepared using a secondary source.
- All CCV criteria were met.
- All interference check (ICSA/ICSAB) criteria were met, with one exception. Cadmium and nickel were detected above the reporting limit in all three ICSA samples. These metals are verified trace impurities in the ICSA standard used by TestAmerica. Therefore, no corrective action was necessary.
- The DT was performed on sample BFB-C-SS-03-03. The DT was only applicable for those metals detected in the parent sample at a concentration of

100x the MDL or greater. All analytes met criteria in the DT, except for those in bold below:

Metal	%D	Criteria
<b>Arsenic</b>	<b>13</b>	%D ≤ 10
Barium	0.9	
<b>Cobalt</b>	<b>14</b>	
Lead	2.1	
Manganese	6.9	
Vanadium	10	

- The PDS for soil was analyzed on the same sample as the DT. The PDS was only applicable for the metals in bold above and those metals not listed above. All metals met criteria in the PDS as follows:

Metal	%R	Criteria
Antimony	95	75-125%
Arsenic	84	
Beryllium	91	
Cadmium	92	
Chromium	86	
Cobalt	83	
Copper	81	
Molybdenum	96	
Nickel	83	
Selenium	82	
Silver	91	
Thallium	93	
Zinc	78	

One method blank and several calibration blanks were analyzed in association with the total ICP/MS analyses in this SDG. All blanks were compliant.

### Completeness

Completeness has been evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All total ICP/MS results for the samples in this SDG were considered usable. Therefore, the completeness for the ICP/MS portion of this SDG is 100%, which meets the minimum acceptance criteria of 95%.

### MERCURY

**General**

The mercury portion of this SDG consisted of eleven (11) soil samples collected on June 25 and 26, 2007. The samples were analyzed for mercury.

The mercury analyses were performed using USEPA SW846 Method 7471A. The samples were analyzed following the procedures outlined in the Work Plan. All samples were prepared and analyzed within the holding time required by the method and the Work Plan.

The samples for mercury were analyzed in one batch (#7186508) under a single ICAL.

**Accuracy**

Accuracy was evaluated using the percent recovery obtained from the LCS samples and the MS/MSD samples. Sample BFB-C-SS-03-03 was designated for MS/MSD analyses for this SDG.

All LCS and MS/MSD recoveries were within acceptance criteria.

**Precision**

Precision was evaluated using the RPD obtained from the MS/MSD concentrations. Precision was further assessed by comparing the field duplicate analyte results. Sample BFB-C-SS-03-12 was collected as a field duplicate of sample BFB-C-SS-03-03.

The MS/MSD RPD met criteria.

Mercury was below the RL in both the parent and field duplicate samples.

**Representativeness**

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing the COC procedures to those described in the Work Plan;
- Comparing actual analytical procedures to those described in the Work Plan;
- Evaluating preservation and holding times; and
- Examining laboratory blanks for cross contamination of samples during analysis.

The samples in this SDG were analyzed following the COC and the analytical procedures described in the Work Plan. The samples were prepared and analyzed within the holding times required by the method.

- All initial calibration criteria were met.
- All calibration verification criteria were met.
- All second source verification criteria were met. The ICV sample was prepared using a secondary source.

- A DT was analyzed on sample BFB-C-SS-03-03. The DT was not applicable because mercury was not detected in the parent sample at a concentration of 25 times the MDL or greater.

One method blank and several calibration blanks were associated with the mercury analyses in this SDG. All blanks were compliant.

### **Completeness**

Completeness has been evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All mercury results for the samples in this SDG were considered usable. The completeness for the mercury portion of this SDG is 100%, which meets the minimum acceptance criteria of 95%.

## **COMPARABILITY**

All data was generated using contract-specific standard methods and reported with known data quality, type of analysis, units, etc.

## **DATA USABILITY**

All calculations were spot checked and verified. All data in this SDG are considered usable for the purposes of this project.